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## MEMORANDUM

DATE: 23 December 1998

TO: David Bennett, WAM, U.S. EPA, Region X

FROM: Michelle Turner, Chemist, WESTON, Seattle  
*RM* Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT: Validation of Organotin Data  
Laboratory Batch: K9806404  
Site: Duwamish River

WORK ASSIGNMENT NO: 46-35-0JZZ

WORK ORDER NO.: 4000-019-038-5200-00

DOC. CONTROL NO.: 4000-019-038-AAAK

cc: Bruce Woods, RAP-WAM, U.S. EPA, Region X  
Dena Hughes, Site Manager, WESTON, Seattle (memo only)  
Kevin Mundell-Jackson, Database Management, WESTON

The quality assurance review of six sediment samples, laboratory batch K9806404, collected from the Duwamish River has been completed. The sediment samples were analyzed for organotins by Columbia Analytical Services of Kelso, Washington. Samples were analyzed by gas chromatography with an FPD detector. The samples were numbered:

98384016      98384021      98384022      98384023      98384024  
98384025

### Data Qualifications

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract. The review follows the format described in the *National Functional Guidelines for Organic Data Review* (EPA OSWER Directive 9240.1, February 1994), modified to include specific requirements of analytical methods.

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1. Timeliness

All samples were extracted 42 days after sample collection, exceeding the 7 day holding time criteria in the Sampling and Analysis Plan. However, prior to extraction, samples were stored frozen, thus extending the holding time. Samples were extracted within the 12 month holding time recommended by PSEP for frozen samples.

2. Detection Limits

Detection limits met project required quantitation limits with the following exceptions

Sample	Compound	QL Goal (µg/Kg)	Reported QL (µg/Kg)
98384016	Tetrabutyltin	10	15
98384016	n-Butyltin	10	30

Where quantitation limit goals were exceeded, undetected analytes were qualified (UI) to indicate matrix interference

3 Initial Calibration

A seven-point initial calibration was performed prior to each analytical batch. The percent relative standard deviation for the initial calibration was within limits of less than 25 percent RSD.

4 Continuing Calibrations

Continuing calibration check was performed after every 10 samples. All target analytes were within required limits for the continuing calibrations with the percent difference for a mid-range standard less than 25 percent.

5. Blanks

a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met. No target analytes were reported in laboratory method blanks.



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b) Field Blanks

No field blanks were associated with this SDG.

6. Surrogate Compound Recovery

Surrogate recovery goals for Tripropyltin were established in the project Sampling and Analysis Plan at 60 to 120 percent for sediment. Based on conversations with the laboratory an additional surrogate, Tripentyltin was added and historical laboratory control chart limits were also used for data qualification. Laboratory limits are presented below:

Surrogate Compound	Sediment Limits
Tripropyltin	18 – 125%
Tripentyltin	28 – 122%

Surrogate compound percent recoveries were within QC limits for all samples.

7. Laboratory Control Sample (LCS)

LCS recovery goals for Butyltins were established in the project Sampling and Analysis Plan at 60 to 130% for sediment. Based on conversations with the laboratory, historical control chart limits of 8 to 161 percent for Dibutyltin and n-Butyltin, and 27 to 162 percent for Tetrabutyltin and Tributyltin were also used for data qualification.

Laboratory control sample percent recoveries met QC guidelines (P-project, L-laboratory), with the following exceptions:

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LCS	Analyte	Percent Recovery	QC Limit	Associated Samples
K981027-LCS	Dibutyltin	45	60-130 (P) 8-161 (L)	98384016
K981027-LCS	n-Butyltin	18	60-130 (P) 8-161 (L)	98384016
K981029-LCS	n-Butyltin	56	60-130 (P) 8-161 (L)	98384021 through 98384025
K981029-DLCS	n-Butyltin	32	60-130 (P) 8-161 (L)	98384021 through 98384025

A LCS/LCS Duplicate was analyzed with this SDG. Relative percent differences (RPDs) between the LCS and LCSD were less than 35 percent, with the exception of the following.

Analyte	LCS %R	LCSD %R	RPD	Associated Samples
K981029-LCS	56	32	56	98384021 through 98384025

Sample results were qualified as estimated (J) when LCS recoveries were outside project limits. Undetected results were also qualified as estimated (UJ) when LCS recoveries were outside project limits

## 8 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

The following matrix spike recovery goals were established in the project Sampling and Analysis Plan at for sediment

Analyte	% Recovery
Tributyltin	40 - 120%
Dibutyltin	30 - 120%
n-Butyltin	10 - 120%



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MS/MSD sample percent recoveries met QC guidelines (P-project, L-laboratory), with the exception of the following:

Sample	Compound	Percent Recovery	QC Limits
98384016MS	Dibutyltin	16	30-120 (P) 8-144 (L)
98384016DMS	Dibutyltin	20	30-120 (P) 8-144 (L)

MS and MSD recoveries for n-Butyltin were not calculated due to matrix interferences

Relative percent differences (RPD) between the MS and MSD percent recoveries met QC guidelines for compounds. No action was based solely on MS/MSD data

9. Field Duplicate Analysis

No field duplicates were associated with this SDG

10. Sample Analysis

A cursory review of raw data was performed. Deliverables were accurate and complete. A duplicate analysis was performed on sample 98384024; no analytes were detected in the sample or duplicate. The case narrative noted that the MS/DMS recovery of n-Butyltin for sample 98384016 was not calculated because of matrix interference. The chromatogram showed components that prevented accurate quantitation of n-Butyltin. Also, the RPD for the LCS/DLCS extracted on 10/29/98 was high for n-Butyltin. This is due to the poor extraction efficiency of this analyte. As LCS and DLCS recoveries were within laboratory QC limits, no further action was taken. No other problems were noted in the case narrative.

11. Laboratory Contact

No laboratory contact was required.

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#### Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values

#### Data Qualifiers

- U - The compound was analyzed for, but was not detected
- UJ - The compound was analyzed for, but was not detected. The associated quantitation limit is an estimate because quality control criteria were not met.
- J - The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less than the quantitation limit or lowest calibration standard.
- R - Quality control indicates that data are unusable (compound may or may not be present) Resampling and reanalysis are necessary for verification
- N - Presumptive evidence of presence of material (tentative identification)
- I - Elevated reporting limit due to matrix interference.

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

Client: Roy F Weston, Inc  
Project: Duwamish River/4000-027-001-2019-38  
Sample Matrix: Sediment

Service Request: K9806404  
Date Collected: 9/15/98  
Date Received: 9/16/98

## Butyltins

Sample Name 98384016  
Lab Code K9806404-007  
Test Notes D

Units: ug/Kg (ppb)  
Basis Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	15	5	10/27/98	10/31/98	ND	UI
Tri-n-butyltin	Method	Butyltins	5	5	10/27/98	10/31/98	ND	
Di-n-butyltin	Method	Butyltins	5	5	10/27/98	10/31/98	ND	UI
n-Butyltin	Method	Butyltins	30	5	10/27/98	10/31/98	ND	UI J

D The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By

*D. Wegel*

Date

*11/11/98*

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

Client: Roy F. Weston, Inc  
Project: Duwamish River/4000-027-001-2019-38  
Sample Matrix: Sediment

Service Request: K9806404  
Date Collected: 9/16/98  
Date Received: 9/17/98

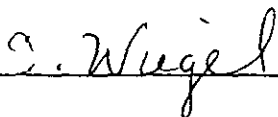
## Butyltins

Sample Name 98384021  
Lab Code: K9806404-012  
Test Notes:

Units: ug/Kg (ppb)  
Basis: Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	3	1	10/29/98	11/1/98	ND	
Tri-n-butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	
Di-n-butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	
n-Butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	UJ

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

Client: Roy F. Weston, Inc  
Project: Duwamish River/4000-027-001-2019-38  
Sample Matrix: Sediment

Service Request: K9806404  
Date Collected: 9/16/98  
Date Received: 9/17/98

## Butyltins

Sample Name 98384022  
Lab Code: K9806404-013  
Test Notes:

Units: ug/Kg (ppb)  
Basis: Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	3	1	10/29/98	11/1/98	ND	
Tri-n-butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	
Di-n-butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	
n-Butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	WJ

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*A. Wiegand*

Date

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

Client: Roy F. Weston, Inc.  
Project: Duwamish River/4000-027-001-2019-38  
Sample Matrix: Sediment

Service Request: K9806404  
Date Collected: 9/16/98  
Date Received: 9/17/98

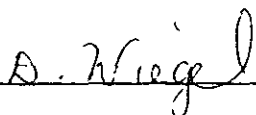
## Butyltins

Sample Name: 98384023  
Lab Code: K9806404-014  
Test Notes:

Units: ug/Kg (ppb)  
Basis: Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	3	1	10/29/98	11/1/98	ND	
Tri-n-butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	
Di-n-butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	
n-Butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	WJ

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

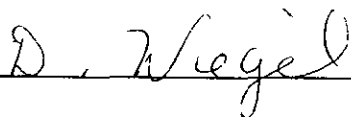
**Service Request:** K9806404  
**Date Collected:** 9/16/98  
**Date Received:** 9/17/98

## Butyltins

**Sample Name** 98384024 **Units** ug/Kg (ppb)  
**Lab Code** K9806404-015 **Basis** Dry  
**Test Notes**

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	3	1	10/29/98	11/1/98	ND	
Tri-n-butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	
Di-n-butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	
n-Butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	WJ

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**COLUMBIA ANALYTICAL SERVICES, INC.**

**Analytical Report**

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9806404  
**Date Collected:** 9/16/98  
**Date Received:** 9/17/98

**Butyltins**

**Sample Name** 98384025 **Units:** ug/Kg (ppb)  
**Lab Code:** K9806404-016 **Basis:** Dry  
**Test Notes**

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	3	1	10/29/98	11/1/98	ND	
Tri-n-butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	
Di-n-butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	
n-Butyltin	Method	Butyltins	1	1	10/29/98	11/1/98	ND	UJ

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*D. Weigel*

Date

*11/11/98*

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*11/12/98*

## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

Client: Roy F Weston, Inc  
Project: Duwamish River/4000-027-001-2019-38  
Sample Matrix: Sediment

Service Request: K9806404  
Date Collected: NA  
Date Received: NA

## Butyltins

Sample Name: Method Blank  
Lab Code: K981027-MB  
Test Notes:

Units: ug/Kg (ppb)  
Basis: Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	3	1	10/27/98	10/31/98	ND	
Tri-n-butyltin	Method	Butyltins	1	1	10/27/98	10/31/98	ND	
Di-n-butyltin	Method	Butyltins	1	1	10/27/98	10/31/98	ND	
n-Butyltin	Method	Butyltins	1	1	10/27/98	10/31/98	ND	

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*E. Wegel*

Date

*11/11/98*

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*10/12/14/98*